



## **Analytical Laboratory**

13339 Hagers Ferry Road Huntersville, NC 28078-7929 McGuire Nuclear Complex - MG03A2 Phone: 980-875-5245 Fax: 980-875-4349

## **Order Summary Report**

Order Number:	J11120115			
Customer Name(s):	Bill Kennedy, Melonie Mart	in, Wayne Chapman, Tom Johnson		
Customer Address:	3195 Pine Hall Rd			
	Mailcode: Belews Steam S	tation		
	Belews Creek, NC 28012			
Lab Contact:	Jason C Perkins	Phone: 980-875-5348		
Report Authorized By: (Signature)		Date:	1/4/2012	

### **Program Comments:**

Please contact the Program Manager (Jason C Perkins) with any questions regarding this report.

#### **Data Flags & Calculations:**

Any analytical tests or individual analytes within a test flagged with a Qualifier indicate a deviation from the method quality system or quality control requirement. The qualifier description is found at the end of the Certificate of Analysis (sample results) under the qualifiers heading. All results are reported on a dry weight basis unless otherwise noted.

### Data Package:

This data package includes analytical results that are applicable only to the samples described in this narrative. An estimation of the uncertainty of measurement for the results in the report is available upon request. This report shall not be reproduced, except in full, without the written consent of the Analytical Laboratory. Please contact the Analytical laboratory with any questions. The order of individual sections within this report is as follows:

Job Summary Report, Sample Identification, Technical Validation of Data Package, Analytical Laboratory Certificate of Analysis, Analytical Laboratory QC Reports, Sub-contracted Laboratory Results, Customer Specific Data Sheets, Reports & Documentation, Customer Database Entries, Test Case Narratives, Chain of Custody (COC)

#### Certification:

The Analytical Laboratory holds the following State Certifications: North Carolina (DENR) Certificate #248, South Carolina (DHEC) Laboratory ID # 99005. Contact the Analytical Laboratory for definitive information about the certification status of specific methods.

## Sample ID's & Descriptions:

Sample ID	Plant/Station	Collection Date and Time	Collected By	Sample Description
2011026219	BELEWS	14-Dec-11 11:30 AM	DAVID MORRIS	FGD Purge Eff
2011026342	BELEWS	14-Dec-11 11:30 AM	DAVID MORRIS	EQ TANK EFF.
2011026343	BELEWS	14-Dec-11 11:30 AM	DAVID MORRIS	BIOREACTOR 1 INF.
2011026344	BELEWS	14-Dec-11 11:30 AM	DAVID MORRIS	BIOREACTOR 2 INF.
2011026345	BELEWS	14-Dec-11 11:30 AM	DAVID MORRIS	BIOREACTOR 2 EFF.
2011026346	BELEWS	14-Dec-11 11:30 AM	DAVID MORRIS	FILTER BLANK
2011026347	BELEWS	14-Dec-11 11:30 AM	DAVID MORRIS	Trip Blank
2011026356	BELEWS	14-Dec-11 11:40 AM	DAVID MORRIS	BIOREACTOR 1 INF.
2011026357	BELEWS	14-Dec-11 11:40 AM	DAVID MORRIS	HG BLANK BIOREACTOR 1 INF.
2011026358	BELEWS	14-Dec-11 11:50 AM	DAVID MORRIS	BIOREACTOR 2 INF.
2011026359	BELEWS	14-Dec-11 11:50 AM	DAVID MORRIS	Hg Blk BioReactor 2 Inf
2011026360	BELEWS	14-Dec-11 11:45 AM	DAVID MORRIS	BIOREACTOR 2 EFF.
2011026361	BELEWS	14-Dec-11 11:45 AM	DAVID MORRIS	Hg Blk BioReactor 2 Eff

## **Technical Validation Review**

### **Checklist:**

Mary Ann Ogle

Reviewed By:

		COC and .pdf report are in agreement with sample and analyses (compliance programs and procedure		<b>✓</b> Yes	☐ No
		All Results are less than the laboratory reporting lim	its.	Yes	<b>✓</b> No
		All laboratory QA/QC requirements are acceptable.		<b>✓</b> Yes	☐ No
		The Vendor Laboratories have been qualified by the Analytical Laboratory	)	Yes	
Repo	rt S	ections Included:			
	<b>✓</b> Jo	b Summary Report	✓ Sub-d	contracted Labora	tory Results
	<b>✓</b> Sa	imple Identification	☐ Custo	omer Specific Data	a Sheets, Reports, & Documentation
	<b>✓</b> Te	chnical Validation of Data Package	☐ Custo	omer Database Er	ntries
	<b>✓</b> Ar	alytical Laboratory Certificate of Analysis	✓ Chair	of Custody	
	☐ Ar	alytical Laboratory QC Report	<b>✓</b> Elect	ronic Data Delive	rable (EDD) Sent Separately

Date:

1/4/2012

## **Certificate of Laboratory Analysis**

This report shall not be reproduced, except in full.

### Order # J11120115

Site: FGD Purge Eff Sample #: 2011026219

Collection Date: 14-Dec-11 11:30 AM Matrix: OTHER

Analyte	Result	Units	Qualifiers	RDL	Method	Analysis Date/Time	Analyst
INORGANIC IONS BY IC							
Bromide	84	mg/L		5	EPA 300.0	19-Dec-11 19:30	JAHERMA
MERCURY (COLD VAPOR) II	N WATER						
Mercury (Hg)	229	ug/L		5	EPA 245.1	16-Dec-11 09:09	AGIBBS
TOTAL RECOVERABLE MET	TALS BY ICD						
Boron (B)	146	mg/L		0.5	EPA 200.7	16-Dec-11 10:17	DJSULL1
,							
DISSOLVED METALS BY ICE							
Selenium (Se)	419	ug/L		10	EPA 200.8	20-Dec-11 10:55	MHH7131
TOTAL RECOVERABLE MET	TALS BY ICP-MS						
Arsenic (As)	170	ug/L		10	EPA 200.8	21-Dec-11 09:43	MHH7131
Chromium (Cr)	172	ug/L		10	EPA 200.8	21-Dec-11 09:43	MHH7131
Copper (Cu)	108	ug/L		10	EPA 200.8	21-Dec-11 09:43	MHH7131
Nickel (Ni)	166	ug/L		10	EPA 200.8	21-Dec-11 09:43	MHH7131
Selenium (Se)	4440	ug/L		10	EPA 200.8	21-Dec-11 09:43	MHH7131
Silver (Ag)	< 10.0	ug/L		10	EPA 200.8	21-Dec-11 09:43	MHH7131
Zinc (Zn)	181	ug/L		20	EPA 200.8	21-Dec-11 09:43	MHH7131
SELENIUM SPECIATION							
Vendor Parameter	Complet	e			V_AS&C		
TOTAL DISSOLVED SOLIDS	į						
TDS	15000	mg/L		200	SM2540C	19-Dec-11 14:36	AGIBBS
0'' 50 TANK 555						"	

Site: EQ TANK EFF. Sample #: 2011026342

Collection Date: 14-Dec-11 11:30 AM Matrix: OTHER

Analyte	Result	Units	Qualifiers	RDL	Method	Analysis Date/Time	Analyst
MERCURY (COLD VAPOR) IN WA	<u>TER</u>						
Mercury (Hg)	208	ug/L		2.5	EPA 245.1	16-Dec-11 09:12	AGIBBS
TOTAL RECOVERABLE METALS	BY ICP						
Boron (B)	152	mg/L		0.5	EPA 200.7	16-Dec-11 10:21	DJSULL1
DISSOLVED METALS BY ICP-MS							
Selenium (Se)	180	ug/L		10	EPA 200.8	20-Dec-11 10:55	MHH7131

2011026342

## **Certificate of Laboratory Analysis**

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### Order # J11120115

Site: EQ TANK EFF. Sample #:

Collection Date: 14-Dec-11 11:30 AM Matrix: OTHER

Analyte	Result	Units	Qualifiers	RDL	Method	Analysis Date/Time	Analyst
TOTAL RECOVERABLE METALS E	BY ICP-MS						
Arsenic (As)	164	ug/L		10	EPA 200.8	21-Dec-11 09:46	MHH7131
Chromium (Cr)	169	ug/L		10	EPA 200.8	21-Dec-11 09:46	MHH7131
Copper (Cu)	106	ug/L		10	EPA 200.8	21-Dec-11 09:46	MHH7131
Nickel (Ni)	162	ug/L		10	EPA 200.8	21-Dec-11 09:46	MHH7131
Selenium (Se)	4160	ug/L		10	EPA 200.8	21-Dec-11 09:46	MHH7131
Silver (Ag)	< 10.0	ug/L		10	EPA 200.8	21-Dec-11 09:46	MHH7131
Zinc (Zn)	178	ug/L		20	EPA 200.8	21-Dec-11 09:46	MHH7131

Site: BIOREACTOR 1 INF. Sample #: 2011026343

Collection Date: 14-Dec-11 11:30 AM Matrix: OTHER

Analyte	Result	Units	Qualifiers	RDL	Method	Analysis Date/Time	Analyst			
TOTAL RECOVERABLE METALS B	SY ICP									
Boron (B)	146	mg/L		0.5	EPA 200.7	16-Dec-11 10:25	DJSULL1			
DISSOLVED METALS BY ICP-MS										
Selenium (Se)	141	ug/L		10	EPA 200.8	20-Dec-11 10:55	MHH7131			
TOTAL RECOVERABLE METALS BY ICP-MS										
Arsenic (As)	< 10.0	ug/L		10	EPA 200.8	21-Dec-11 09:49	MHH7131			
Chromium (Cr)	< 10.0	ug/L		10	EPA 200.8	21-Dec-11 09:49	MHH7131			
Copper (Cu)	< 10.0	ug/L		10	EPA 200.8	21-Dec-11 09:49	MHH7131			
Nickel (Ni)	39.7	ug/L		10	EPA 200.8	21-Dec-11 09:49	MHH7131			
Selenium (Se)	133	ug/L		10	EPA 200.8	21-Dec-11 09:49	MHH7131			
Silver (Ag)	< 10.0	ug/L		10	EPA 200.8	21-Dec-11 09:49	MHH7131			
Zinc (Zn)	< 20.0	ug/L		20	EPA 200.8	21-Dec-11 09:49	MHH7131			
SELENIUM SPECIATION										
Vendor Parameter	Complete	)			V_AS&C					

Site: BIOREACTOR 2 INF. Sample #: 2011026344

Collection Date: 14-Dec-11 11:30 AM Matrix: OTHER

Analyte	Result	Units	Qualifiers	RDL	Method	Analysis Date/Time	Analyst
TOTAL RECOVERABLE METALS	BY ICP						
Boron (B)	138	mg/L		0.5	EPA 200.7	16-Dec-11 10:29	DJSULL1

## **Certificate of Laboratory Analysis**

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### Order # J11120115

Site: BIOREACTOR 2 INF.

Sample #:

2011026344

Collection Date: 14-Dec-11 11:30 AM

Matrix:

OTHER

Analyte	Result	Units	Qualifiers	RDL	Method	Analysis Date/Time	Analyst
TOTAL RECOVERABLE METAL	S BY ICP-MS						
Arsenic (As)	< 10.0	ug/L		10	EPA 200.8	21-Dec-11 09:52	MHH7131
Chromium (Cr)	< 10.0	ug/L		10	EPA 200.8	21-Dec-11 09:52	MHH7131
Copper (Cu)	< 10.0	ug/L		10	EPA 200.8	21-Dec-11 09:52	MHH7131
Nickel (Ni)	< 10.0	ug/L		10	EPA 200.8	21-Dec-11 09:52	MHH7131
Selenium (Se)	14.5	ug/L		10	EPA 200.8	21-Dec-11 09:52	MHH7131
Silver (Ag)	< 10.0	ug/L		10	EPA 200.8	21-Dec-11 09:52	MHH7131
Zinc (Zn)	< 20.0	ug/L		20	EPA 200.8	21-Dec-11 09:52	MHH7131

Site: BIOREACTOR 2 EFF.

Sample #:

2011026345

Collection Date: 14-Dec-11 11:30 AM

Matrix:

**OTHER** 

Analyte	Result	Units	Qualifiers	RDL	Method	Analysis Date/Time	Analyst			
INORGANIC IONS BY IC										
Bromide	82	mg/L		5	EPA 300.0	19-Dec-11 19:46	JAHERMA			
MERCURY (COLD VAPOR) IN WAT	ΓER									
Mercury (Hg)	< 1.00	ug/L		1	EPA 245.1	16-Dec-11 09:14	AGIBBS			
TOTAL RECOVERABLE METALS BY ICP										
Boron (B)	129	mg/L		0.5	EPA 200.7	16-Dec-11 10:32	DJSULL1			
TOTAL RECOVERABLE METALS BY ICP-MS										
Arsenic (As)	< 5.00	ug/L		5	EPA 200.8	21-Dec-11 09:55	MHH7131			
Chromium (Cr)	< 5.00	ug/L		5	EPA 200.8	21-Dec-11 09:55	MHH7131			
Copper (Cu)	< 5.00	ug/L		5	EPA 200.8	21-Dec-11 09:55	MHH7131			
Nickel (Ni)	< 5.00	ug/L		5	EPA 200.8	21-Dec-11 09:55	MHH7131			
Selenium (Se)	< 5.00	ug/L		5	EPA 200.8	21-Dec-11 09:55	MHH7131			
Silver (Ag)	< 5.00	ug/L		5	EPA 200.8	21-Dec-11 09:55	MHH7131			
Zinc (Zn)	< 10.0	ug/L		10	EPA 200.8	21-Dec-11 09:55	MHH7131			
SELENIUM SPECIATION										
Vendor Parameter	Complete	е			V_AS&C					

Site: FILTER BLANK

Sample #:

2011026346

Collection Date: 14-Dec-11 11:30 AM

Matrix:

OTHER

Analyte	Result	Units	Qualifiers	RDL	Method	Analysis Date/Time	Analyst
DISSOLVED METALS BY ICP-MS							
Selenium (Se)	< 1.000	ug/L		1	EPA 200.8	20-Dec-11 10:55	MHH7131

## **Certificate of Laboratory Analysis**

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### Order # J11120115

Site: Trip Blank Sample #: 2011026347 OTHER Collection Date: 14-Dec-11 11:30 AM Matrix: Analyte Result Units Qualifiers **RDL** Method Analysis Date/Time **Analyst TOTAL RECOVERABLE METALS BY ICP** Boron (B) 0.05 DJSULL1 < 0.050 mg/L EPA 200.7 16-Dec-11 10:13 **TOTAL RECOVERABLE METALS BY ICP-MS** Arsenic (As) < 1.000 ug/L 1 EPA 200.8 21-Dec-11 09:40 MHH7131 Chromium (Cr) < 1.000 ug/L **EPA 200.8** 21-Dec-11 09:40 MHH7131 1 Copper (Cu) < 1.000 ug/L EPA 200.8 21-Dec-11 09:40 MHH7131 Nickel (Ni) < 1.000 ug/L 1 EPA 200.8 21-Dec-11 09:40 MHH7131 Selenium (Se) ug/L **EPA 200.8** 21-Dec-11 09:40 MHH7131 < 1.000 1 Silver (Ag) < 1.000 ug/L EPA 200.8 21-Dec-11 09:40 MHH7131 < 2.00 ug/L 2 EPA 200.8 21-Dec-11 09:40 MHH7131 Zinc (Zn) **SELENIUM SPECIATION** Vendor Parameter Complete V\_AS&C Site: BIOREACTOR 1 INF. Sample #: 2011026356 Collection Date: 14-Dec-11 11:40 AM Matrix: OTHER Qualifiers RDL Method Analyte Result Units Analysis Date/Time **Analyst MERCURY 1631** Vendor Parameter Complete V\_BRAND Site: HG BLANK BIOREACTOR 1 INF. Sample #: 2011026357 Collection Date: 14-Dec-11 11:40 AM Matrix: **OTHER** Analyte Result Units Qualifiers **RDL** Method **Analysis Date/Time Analyst MERCURY 1631** Vendor Parameter V\_BRAND Complete Site: BIOREACTOR 2 INF. Sample #: 2011026358 Collection Date: 14-Dec-11 11:50 AM Matrix: **OTHER** Analyte Result Units Qualifiers **RDL** Method Analysis Date/Time **Analyst** MERCURY 1631 Vendor Parameter Complete V\_BRAND

Analyst

Analysis Date/Time

### **Certificate of Laboratory Analysis**

This report shall not be reproduced, except in full.

### Order # J11120115

Site: Hg Blk BioReactor 2 Inf

Collection Date: 14-Dec-11 11:50 AM

Sample #: 2011026359

Matrix: OTHER

Qualifiers

**RDL** 

Method

MERCURY 1631

Analyte

Vendor Parameter Complete V\_BRAND

Units

Result

Site: BIOREACTOR 2 EFF. Sample #: 2011026360

Collection Date: 14-Dec-11 11:45 AM Matrix: OTHER

Analyte Result Units Qualifiers RDL Method Analysis Date/Time Analyst

MERCURY 1631

Vendor Parameter Complete V\_BRAND

Site: Hg Blk BioReactor 2 Eff Sample #: 2011026361

Collection Date: 14-Dec-11 11:45 AM Matrix: OTHER

Analyte Result Units Qualifiers RDL Method Analysis Date/Time Analyst

MERCURY 1631

Vendor Parameter Complete V\_BRAND



18804 Northcreek Parkway Bothell, WA, 98011 Tel: (425) 483-3300 Fax: (425) 483-9818 www.appliedspeciation.com

December 22, 2011

Jay Perkins Duke Energy Analytical Laboratory Mail Code MGO3A2 (Building 7405) 13339 Hagers Ferry Rd. Huntersville, NC 28078 (704) 875-5245

Project: Belews – FGD WWTS Bi-Monthly Sampling) (LIMS # J11120115)

Dear Mr. Perkins,

Attached is the report associated with four (4) aqueous samples submitted for selenium speciation analysis on December 15, 2011. The samples were received in a sealed cooler at -0.7°C on December 16, 2011. Selenium speciation analysis was performed via ion chromatography inductively coupled plasma dynamic reaction cell mass spectrometry (IC-ICP-DRC-MS). Any issues associated with the analysis are addressed in the following report.

If you have any questions, please feel free to contact me at your convenience.

Sincerely,

Russell Gerads Vice President

Applied Speciation and Consulting, LLC

### Applied Speciation and Consulting, LLC

Report prepared for:

Jay Perkins Duke Energy Analytical Laboratory Mail Code MGO3A2 (Building 7405) 13339 Hagers Ferry Rd. Huntersville, NC 28078

Project: Belews – FGD WWTS Bi-Monthly Sampling) (LIMS # J11120115)

December 22, 2011

### 1. Sample Reception

Four (4) aqueous samples in 125mL HDPE bottles (provided by Applied Speciation and Consulting) were submitted for selenium speciation analysis on December 15, 2011. The samples were received on December 16, 2011 in a sealed container at -0.7°C.

The samples were received in a laminar flow clean hood, void of trace metals contamination and ultra-violet radiation, and was designated a discrete sample identifier. An aliquot of each sample was filtered (0.45 µm) and each filtrate was stored in a secure, monitored cryofreezer (maintained at a temperature of -80 °C) until selenium speciation analysis could be performed via ion chromatography inductively coupled plasma dynamic reaction cell mass spectrometry (IC-ICP-DRC-MS).

### 2. Sample Preparation

All sample preparation is performed in laminar flow clean hoods known to be free from trace metals contamination. All applied water for dilutions and sample preservatives are monitored for contamination to account for any biases associated with the sample results.

<u>Selenium Speciation Analysis by IC-ICP-DRC-MS</u> Prior to analysis, an aliquot of each sample was filtered with a syringe filter (0.45µm) and injected directly into a sealed autosampler vial. No further sample preparation was performed as any chemical alteration of a sample may shift the equilibrium of the system, resulting in changes in speciation ratios.

### 3. Sample Analysis

All sample analysis is preceded by a minimum of a five-point calibration curve spanning the entire concentration range of interest. Calibration curves are performed at the beginning of

each analytical day. All calibration curves, associated with each species of interest, are standardized by linear regression resulting in a response factor. All sample results are **instrument blank corrected** to account for any operational biases associated with the analytical platform.

Prior to sample analysis, all calibration curves are verified using second source standards which are identified as initial calibration verification standards (ICV).

Ongoing instrument performance is identified by the analysis of continuing calibration verification standards (CCV) and continuing calibration blanks (CCB) at a minimal interval of every ten analytical runs.

<u>Selenium Speciation Analysis by IC-ICP-DRC-MS</u> Each sample for selenium speciation analysis was analyzed by ion chromatography inductively coupled plasma dynamic reaction cell mass spectrometry (IC-ICP-DRC-MS) on December 16, 2011. An aliquot of each sample is injected onto an anion exchange column and mobilized by a basic (pH > 7) gradient. The eluting selenium species are then introduced into a radio frequency (RF) plasma where energy-transfer processes cause desolvation, atomization, and ionization. The ions are extracted from the plasma through a differentially-pumped vacuum interface and travel through a pressurized chamber (DRC) containing a specific reactive gas which preferentially reacts with interfering ions of the same target mass to charge ratios (m/z). A solid-state detector detects ions transmitted through the mass analyzer and the resulting current is processed by a data handling system.

Retention times for each eluting species are compared to known standards for species identification.

### 4. Analytical Issues

The overall analyses went well and no significant analytical issues were encountered. All quality control parameters associated with this sample were within acceptance limits.

The estimated method detection limits (eMDLs) for selenite, selenate, and selenocyanate are generated from replicate analyses of the lowest standard in the calibration curve. Not all selenium species are present in preparation blanks; therefore, eMDL calculations based on preparation blanks are artificially biased low.

The eMDL for methylseleninic acid and selenomethionine is calculated from the average eMDL of selenite, selenate, and selenocyanate. The calibration does not contain methylseleninic acid or selenomethionine due to impurities in these standards which would bias the results for other selenium species.

If you have any questions or concerns regarding this report, please feel free to contact me.

Sincerely,

Russell Gerads Vice President

Applied Speciation and Consulting, LLC

# Selenium Speciation Results for Duke Energy Project Name: Belews - FGD WWTS Bi-Monthly Sampling) Contact: Jay Perkins LIMS #J11120115

Date: December 22, 2011 Report Generated by: Russell Gerads Applied Speciation and Consulting, LLC

### Sample Results

Sample ID	Se(IV)	Se(VI)	SeCN	MeSe(IV)	SeMe	Unknown Se Species (n)
FGD Purge Eff	54.1	85.8	7.6	ND (<1.9)	ND (<1.9)	0 (0)
BioReactor 1 Inf	19.3	92.0	ND (<0.39)	ND (<0.47)	ND (<0.47)	2.00 (1)
BioReactor 2 Eff	ND (<0.56)	ND (<0.47)	ND (<0.39)	ND (<0.47)	ND (<0.47)	0 (0)
Metals Trip Blk	ND (<0.11)	ND (<0.093)	ND (<0.078)	ND (<0.094)	ND (<0.094)	0 (0)

All results reflect the applied dilution and are reported in µg/L

ND = Not detected at the applied dilution

SeCN = Selenocyanate

MeSe(IV) = Methylseleninic acid

SeMe = Selenomethionine

Unknown Se Species = Total concentration of all unknown Se species observed by IC-ICP-MS

n = number of unknown Se species observed

### Selenium Speciation Results for Duke Energy Project Name: Belews - FGD WWTS Bi-Monthly Sampling) Contact: Jay Perkins LIMS #J11120115

Date: December 22, 2011 Report Generated by: Russell Gerads Applied Speciation and Consulting, LLC

### **Quality Control Summary - Preparation Blank Summary**

Analyte (µg/L)	PBW1	PBW2	PBW3	PBW4	Mean	StdDev	eMDL*	eMDL 10x	eMDL 50x	eMDL 200x
Se(IV)	0.000	0.000	0.000	0.000	0.000	0.000	0.011	0.11	0.56	2.2
Se(VI)	0.000	0.000	0.000	0.000	0.000	0.000	0.009	0.093	0.47	1.9
SeCN	0.000	0.000	0.000	0.000	0.000	0.000	0.008	0.078	0.39	1.6
MeSe(IV)	0.000	0.000	0.000	0.000	0.000	0.000	0.009	0.094	0.47	1.9
SeMe	0.000	0.000	0.000	0.000	0.000	0.000	0.009	0.094	0.47	1.9

eMDL = Estimated Method Detection Limit

### **Quality Control Summary - Certified Reference Materials**

Analyte (µg/L)	CRM	True Value	Result	Recovery
Se(IV)	LCS	3.83	4.09	106.8
Se(VI)	LCS	3.79	4.18	110.1
SeCN	LCS	3.57	3.83	107.3
MeSe(IV)	LCS	2.59	2.52	97.4
SeMe	LCS	3.73	3.77	101.1

<sup>\*</sup>Please see narrative regarding eMDL calculations

# Selenium Speciation Results for Duke Energy Project Name: Belews - FGD WWTS Bi-Monthly Sampling) Contact: Jay Perkins LIMS #J11120115

Date: December 22, 2011
Report Generated by: Russell Gerads
Applied Speciation and Consulting, LLC

### **Quality Control Summary - Matrix Duplicates**

Analyte (µg/L)	Sample ID	Rep 1	Rep 2	Mean	RPD
Se(IV)	BioReactor 2 Eff	ND (<0.56)	ND (<0.56)	NC	NC
Se(VI)	BioReactor 2 Eff	ND (<0.47)	ND (<0.47)	NC	NC
SeCN	BioReactor 2 Eff	ND (<0.39)	ND (<0.39)	NC	NC
MeSe(IV)	BioReactor 2 Eff	ND (<0.47)	ND (<0.47)	NC	NC
SeMe	BioReactor 2 Eff	ND (<0.47)	ND (<0.47)	NC	NC

ND = Not detected at the applied dilution

NC = Value was not calculated due to one or more concentrations below the eMDL

### **Quality Control Summary - Matrix Spike/ Matrix Spike Duplicate**

Analyte (µg/L)	Sample ID	Spike Conc	MS Result	Recovery	Spike Conc	MSD Result	Recovery	RPD
Se(IV)	BioReactor 2 Eff	278.0	266.0	95.7	278.0	261.6	94.1	1.6
Se(VI)	BioReactor 2 Eff	252.3	252.5	100.1	252.3	251.7	99.8	0.3
SeCN	BioReactor 2 Eff	228.8	211.8	92.6	228.8	213.0	93.1	0.6

Page 16 of 28 2) Client: 8)Oper. Unit: 5)Business Unit: )Project Name LAB USE ONLY ..... B)Seal/Locked By )Relinquished By i)Seal/Locked By Energy.. Customer to complete appropriate columns to right Wayne Chapman, Tom Johnson WWTS Bi-Monthly Sampling) Se Speciation Bottle Bill Kennedy, Melonie Martin, omer to sign & date below - fill out from left to right ₽ Belews - FGD CHAIN OF CUSTODY RECORD AND ANALYSIS REQUEST FORM 6)Process: 9)Res. Type: Duke Energy Analytical Laboratory Mall Code MGO3A2 (Bullding 7405) <sup>13</sup>Sample Description or ID Huntersville, N. C. 28078 (704) 875-5245 C. N. Se Ao. Zn by IMS 13339 Hagers Ferry Rd Fax: (704) 875-4349 \_\_\_ BioReactor 2 Eff BioReactor 2 Inf BioReactor 1 Inf FGD Purge Eff EQ Tank Eff. Date/Time Date/Time Metals Trip Blk 4)Fax No: Filter BK 10)Reso. Center: Mail Code: Digestions = TRM (0) Seal/Lock Opened By appropriate non-shaded areas. 12)Seal/Lock Opened By Sampling conducted: 2nd and 4th Wednesday 12/14 AS&C 12/14 2/14 Date PO#133241 Customer to complete all 2 - T 08:13 1 30 13:30 <u>ئ</u> હ્યુ 3 8 thomas.d.johnson@siemens.com 1 how the <sup>15</sup>Prèserv.:1=HCL 2=H<sub>2</sub>SO<sub>4</sub> 3=HNO<sub>5</sub> المريدة Filtening of the Se is performed in the field please provide a filter blank too. Polled 7Comp. <sup>16</sup>Analyses Required Date/Time <sup>18</sup>Grab Date/Time TDS Samples Originating £ Water Hg - 245.1 SAMPLE PROGRAM Br (Dionex) Metals\* ģ \_ 3,4 Se, soluble (no dig.) Drinking Water Customer, IMPORTANT! Neppens Ground Please indicate desired turnaround. y DISTRIBUTION
ORIGINAL to LAB. <sup>22</sup>Requested Turnaround COPY to CLIENT \*7 Days 14 Days ·48 Hr Add. Cost Will Apply Se, speciation - vendor to AS&C (Important to place filled bottle back into both baggies) Ū 16/1



December 28, 2011

Duke Energy ATTN: Jay Perkins 13339 Hagers Ferry Road Huntersville NC 28078 jperkins@duke-energy.com

RE: Project DUK-HV1101 Client Project: J11120115

Dear Jay Perkins,

This report contains results for the 6 samples received by Brooks Rand Labs (BRL) on December 16, 2011. The samples were logged-in for the contracted analyses according to the chain-of-custody form(s). The samples were received, prepared, analyzed, and stored according to BRL SOPs and EPA methodology.

The results were method blank corrected as described in the calculations section of the relevant BRL SOP(s) and may have been evaluated using reporting limits that have been adjusted to account for sample aliquot size. Please refer to the *Sample Results* page for sample-specific MDLs, MRLs, and other details. All data is reported without qualification (with the exception of concentration qualifiers), and all associated quality control sample results meet the acceptance criteria.

BRL, an accredited laboratory, certifies that the reported results of all analyses for which BRL is NELAP accredited meet all NELAP requirements. For more details, please see the *Report Information* page in your report. Please feel free to contact me if you have any questions regarding this report.

Sincerely,

Tiffany Stilwater Project Manager

tiffany@brooksrand.com



Analytical Lab PageRL8Refp28t 1151039 Client PM: Jay Perkins Client PO: 141391

## Report Information

### **Laboratory Accreditation**

BRL is accredited by the *National Environmental Laboratory Accreditation Program* (NELAP) through the State of Florida Department of Health, Bureau of Laboratories (E87982) and is certified to perform many environmental analyses. BRL is also certified by many other states to perform environmental analyses. For a current list of our accreditations/certifications, please visit our website at <a href="http://www.brooksrand.com/default.asp?contentID=586">http://www.brooksrand.com/default.asp?contentID=586</a>. Results reported relate only to the samples listed in the report.

### **Field Quality Control Samples**

Please be notified that certain EPA methods require the collection of field quality control samples of an appropriate type and frequency; failure to do so is considered a deviation from some methods and for compliance purposes should only be done with the approval of regulatory authorities. Please see the specific EPA methods for details regarding required field quality control samples.

### **Common Abbreviations**

BLK	method blank	MS	matrix spike
BRL	Brooks Rand Labs	MSD	matrix spike duplicate
BS	laboratory fortified blank	ND	non-detect
CAL	calibration standard	NR	non-reportable
CCV	continuing calibration verification	PS	post preparation spike
COC	chain of custody record	REC	percent recovery
CRM	certified reference material	RPD	relative percent difference
D	dissolved fraction	RSD	relative standard deviation
DUP	duplicate	SCV	secondary calibration verification
ICV	initial calibration verification	SOP	standard operating procedure
MDL	method detection limit	SRM	standard reference material
MRL	method reporting limit	Т	total recoverable fraction

### **Definition of Data Qualifiers**

(Effective 9/23/09)

- B Detected by the instrument, the result is > the MDL but ≤ the MRL. Result is reported and considered an estimate.
- **E** An estimated value due to the presence of interferences. A full explanation is presented in the narrative.
- **H** Holding time and/or preservation requirements not met. Result is estimated.
- **J** Estimated value. A full explanation is presented in the narrative.
- J-M Duplicate precision (RPD) for associated QC sample was not within acceptance criteria. Result is estimated.
- J-N Spike recovery for associated QC sample was not within acceptance criteria. Result is estimated.
- **M** Duplicate precision (RPD) was not within acceptance criteria. Result is estimated.
- N Spike recovery was not within acceptance criteria. Result is estimated.
- **R** Rejected, unusable value. A full explanation is presented in the narrative.
- U Result is ≤ the MDL or client requested reporting limit (CRRL). Result reported as the MDL or CRRL.
- X Result is not BLK-corrected and is within 10x the absolute value of the highest detectable BLK in the batch. Result is estimated.

These qualifiers are based on those previously utilized by Brooks Rand, Ltd., those found in the EPA <u>SOW ILM03.0</u>, Exhibit B, Section III, pg. B-18, and the <u>USEPA Laboratory Data Validation Functional Guidelines for Evaluating Inorganic Analyses; USEPA; July 2002. These supersede all previous qualifiers ever employed by BRL.</u>

**Project ID:** DUK-HV1101 **PM:** Tiffany Stilwater



Analytical Lab
Pa@RL9Refp28t 1151039
Client PM: Jay Perkins
Client PO: 141391

# Sample Information

Sample	Lab ID	Report Matrix	Type	Sampled	Received
BioReactor 1 Inf	1151039-01	Influent	Sample	12/14/2011	12/16/2011
Hg Blk BioReactor 1 Inf	1151039-02	DIW	Field Blank	12/14/2011	12/16/2011
BioReactor 2 Inf	1151039-03	Influent	QC Sample	12/14/2011	12/16/2011
Hg Blk BioReactor 2 Inf	1151039-04	DIW	Field Blank	12/14/2011	12/16/2011
BioReactor 2 Eff	1151039-05	Effluent	Sample	12/14/2011	12/16/2011
Hg Blk BioReactor 2 Eff	1151039-06	DIW	Field Blank	12/14/2011	12/16/2011

## **Batch Summary**

Analyte	Lab Matrix	Method	Prepared	Analyzed	Batch	Sequence
Hg	Water	EPA 1631	12/19/2011	12/20/2011	B112132	1100906



Analytical Lab Pa@R20Refp28t 1151039 Client PM: Jay Perkins Client PO: 141391

# Sample Results

Sample	Analyte	Report Matrix	Fraction	Result	Qualifier	MDL	MRL	Unit	Batch	Sequence
<b>BioReactor 1 In</b> 1151039-01	<b>f</b> Hg	Influent	Т	161		1.52	4.04	ng/L	B112132	1100906
<b>BioReactor 2 Et</b> 1151039-05	<b>ff</b> Hg	Effluent	Т	39.4		0.61	1.61	ng/L	B112132	1100906
<b>BioReactor 2 In</b> 1151039-03	<b>f</b> Hg	Influent	Т	102		1.52	4.04	ng/L	B112132	1100906
<b>Hg Blk BioReac</b> 1151039-02	e <b>tor 1 Inf</b> Hg	DIW	Т	0.15	U	0.15	0.40	ng/L	B112132	1100906
<b>Hg Blk BioReac</b> 1151039-06	e <b>tor 2 Eff</b> Hg	DIW	Т	0.15	U	0.15	0.40	ng/L	B112132	1100906
<b>Hg Blk BioRead</b> 1151039-04	e <b>tor 2 Inf</b> Hg	DIW	Т	0.15	U	0.15	0.40	ng/L	B112132	1100906



Analytical Lab Pa@R21Refp28t 1151039 Client PM: Jay Perkins Client PO: 141391

# Accuracy & Precision Summary

Batch: B112132 Lab Matrix: Water Method: EPA 1631

Sample B112132-SRM1	Analyte Certified Reference Materia Hg	Native al (1149037	<b>Spike</b> <b>7, NIST 1641d</b> 15.68	Result 1000x dilut 15.29	Units ion) ng/L	<b>REC &amp; Limits</b> 98% 85-115	RPD & Limits
B112132-MS2	Matrix Spike (1151039-03) Hg	102.3	505.1	664.3	ng/L	111% 71-125	
B112132-MSD2	Matrix Spike Duplicate (115	5 <b>1039-03)</b> 102.3	505.1	648.2	ng/L	108% 71-125	2% 24
B112132-MS3	<b>Matrix Spike (1151039-05)</b> Hg	39.35	177.4	228.6	ng/L	107% 71-125	
B112132-MSD3	Matrix Spike Duplicate (115	5 <b>1039-05)</b> 39.35	180.9	226.1	ng/L	103% 71-125	1% 24

**Project ID:** DUK-HV1101 **PM:** Tiffany Stilwater



Analytical Lab PageR22Refp28t 1151039 Client PM: Jay Perkins Client PO: 141391

# Method Blanks & Reporting Limits

Batch: B112132 Matrix: Water Method: EPA 1631

Analyte: Hg

Sample	Result	Units
B112132-BLK1	0.06	ng/L
B112132-BLK2	0.08	ng/L
B112132-BLK3	0.06	ng/L
B112132-BLK4	0.04	ng/L

 Average: 0.06
 Standard Deviation: 0.02
 MDL: 0.15

 Limit: 0.50
 Limit: 0.10
 MRL: 0.40



Analytical Lab PageR23Rofp26t 1151039 Client PM: Jay Perkins **Client PO: 141391** 

## **Instrument Calibration**

**Sequence:** 1100906 **Total Mercury and Mercury Speciation by CVAFS** Instrument: THG-10

Method: EPA 1631

Date: 12/20/2011 Analyte: Hg

<b>Lab ID</b> 1100906-IBL1	True Value	Result 3.72	Units pg of Hg	REC & Lim	
1100906-IBL2		3.49	pg of Hg		
1100906-IBL3		3.46	pg of Hg		
1100906-IBL4		4.98	pg of Hg		
1100906-CAL1	25.00	24.38	pg of Hg	98%	
1100906-CAL2	100.0	98.22	pg of Hg	98%	
1100906-CAL3	500.0	482.6	pg of Hg	97%	
1100906-CAL4	2500	2687	pg of Hg	107%	
1100906-CAL5	10000	10100	pg of Hg	101%	
1100906-ICV1	1568	1529	pg of Hg	98%	85-115
1100906-CCB1		6.98	pg of Hg		
1100906-CCV1	500.0	532.2	pg of Hg	106%	77-123
1100906-CCV2	500.0	515.4	pg of Hg	103%	77-123
1100906-CCV3	500.0	514.5	pg of Hg	103%	77-123

**Project ID:** DUK-HV1101 **PM:** Tiffany Stilwater



Analytical Lab Pa@R24Refp28t 1151039 Client PM: Jay Perkins Client PO: 141391

# **Sample Containers**

Lab ID: 1151039-01 Sample: BioReactor 1 Inf			Report Matrix: Influent Sample Type: Sample				Collected: 12/14/2011 Received: 12/16/2011		
Des A	Container Bottle FLPE Hg-T	Size 250 mL	<b>Lot</b> 71470160 10	Preservation none	P-Lot n/a	рН	Ship. Cont. Cooler		
	<b>D</b> : 1151039-02 <b>ole:</b> Hg Blk BioReactor 1 Inf		•	rt Matrix: DIW le Type: Field Blank			cted: 12/14/2011 ved: 12/16/2011		
Des A	Container Bottle FLPE Hg-T	Size 250 mL	<b>Lot</b> 71470160 10	Preservation none	P-Lot n/a	рН	Ship. Cont. Cooler		
	<b>D:</b> 1151039-03 <b>ple:</b> BioReactor 2 Inf		•	rt Matrix: Influent le Type: QC Sample			ted: 12/14/2011 ved: 12/16/2011		
Des A	Container Bottle FLPE Hg-T	Size 250 mL	Lot 71470160 10	Preservation none	P-Lot n/a	рН	Ship. Cont. Cooler		
Lab ID: 1151039-04 Sample: Hg Blk BioReactor 2 Inf			Repor Samp			ted: 12/14/2011 ved: 12/16/2011			
Des A	Container Bottle FLPE Hg-T	Size 250 mL	Lot 71470160 10	Preservation none	P-Lot n/a	рН	Ship. Cont. Cooler		
	<b>D:</b> 1151039-05 ple: BioReactor 2 Eff		•	rt Matrix: Effluent le Type: Sample		Collected: 12/14/2011 Received: 12/16/2011			
Des A	Container Bottle FLPE Hg-T	Size 250 mL	Lot 71470160 10	Preservation none	P-Lot n/a	рН	Ship. Cont. Cooler		
	<b>D:</b> 1151039-06 <b>ple:</b> Hg Blk BioReactor 2 Eff		•	rt Matrix: DIW le Type: Field Blank			ted: 12/14/2011 ved: 12/16/2011		
Des A	Container Bottle FLPE Hg-T	Size 250 mL	<b>Lot</b> 71470160 10	Preservation none	<b>P-Lot</b> n/a	рН	Ship. Cont. Cooler		

**Project ID:** DUK-HV1101 **PM:** Tiffany Stilwater



Analytical Lab PageR25Ref28t 1151039 Client PM: Jay Perkins Client PO: 141391

# **Shipping Containers**

Cooler

**Received:** December 16, 2011 9:00 **Tracking No:** 4726 7966 6868 via FedEx

Coolant Type: Ice Temperature: 1.7 °C Description: Cooler
Damaged in transit? No
Returned to client? No

Custody seals present? No Custody seals intact? No COC present? Yes

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